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WATER RENOVATION BY ION EXCHANGE

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by

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INTRODUCTION

The potential shortage of fresh water in the United States is well documented.[1] It is estimated that as soon as 1980, the withdrawals of water for all uses will approximately equal the recoverable streamflow and groundwater supply. Because the point of demand is often far removed from the source of supply and some supplies are available only seasonally, multiple reuse of water must become the rule rather soon.[2]

One source of water which is always available at centers of population is domestic sewage. It is amenable to treatment by the usual biological methods and is gathered for that purpose. Even after secondary treatment, however, it is useful only as irrigation or industrial cooling water, and even for these purposes it has some disadvantages. Methods must be found to upgrade these waters for many uses. Renovated sewage could be used for industrial supplies, recreational use, irrigation and recharge of groundwater, thus freeing existing supplies for municipal use.

It is the purpose of this paper to suggest ion exchange as a method of reclaiming water from sewage and to present results from laboratory studies utilizing chloride cycle anion exchange.

WHAT MUST BE REMOVED FROM SEWAGE

Organic Matter

The pollutants present in domestic sewage can be broadly classed as organic and inorganic pollutants. While the organic matter in sewage constitutes less than 0.1 percent of the total by weight, the variety of materials is large. Most obvious is the organic matter which is the waste of human life. This can largely be removed by conventional sewage treatment. Also present is a large variety of living organisms ranging in size from virus to worms and including bacteria and protozoa. Most of these, but by no means all, are removed with sewage treatment.

There are many other organic materials which are not usually removed in sewage treatment and can be detected in effluents by means of the COD (chemical oxygen demand) test. Some of these materials represent the products of the biological treatment process itself. Other materials are part of a broad range of synthetic chemicals which are resistant to biological attack. The best known of these chemicals are ABS detergents, which cause water of sewage origin to foam. However, there are many others, such as herbicides, insecticides and pesticides.

Inorganic Matter

In addition to that present in a water supply before use, inorganic matter is added to water through use. Some is added directly and some is derived from organic matter which is broken down into inorganic matter by biological treatment. Table 1 shows normal increments of inorganic matter added through one use of water.

TABLE 1

SUMMARY OF INCREMENTS OF COMMON IONS IN DOMESTIC WATER AFTER ONE CYCLE OF WATER USE (3)

Analysis	Normal Increase mg/l
Ca^{++} ($CaCO_3$)	15 - 40
Mg^{++} ($CaCO_3$)	15 - 40
Na^{+}	40 - 70
K^{+}	7 - 15
$SO_4^{=}$	15 - 30
$PO_4^{=}$	20 - 40
Cl^{-}	20 - 50
NO_3^{-}	0 - 18
Total N	20 - 40
Total Alkalinity ($CaCO_3$)	100 - 150
Total Dissolved Solids	100 - 300

Algae Nutrients

All plants need certain minerals for growth. Chief among the needed nutrient or fertilizing elements are nitrogen and phosphorus, although small amounts of other elements are necessary also. If stored in an open reservoir, well purified water, even one meeting the U.S. Public Health Service standard for drinking water, may be fouled by growth of algae. It may contain the necessary nutrients so that algae, utilizing sunlight, may grow and pollute an otherwise satisfactory water. The removal of either nitrogen or phosphorus, therefore, is desirable to prevent the growth of algae.

DESCRIPTION OF ION EXCHANGE

Uses

The standard use for the ion exchange process is in the removal of inorganic ions from water[4] . Most common uses in water treatment are in water softening and de-ionization, but ion exchange is also used for reducing acidity or alkalinity, removal of metallic contaminants, removal of radioactive pollutants and recovery of valuable materials.

Many organic materials exist as charged particles and thus may be removed by ion exchange. Others are adsorbed on an ion exchange resin and are difficult to remove, fouling the resin. An example of a commercial use of ion exchange is the removal of color and ash from sugar solutions by adsorption and ion exchange.

Renovation of Sewage

For normal sewage, the principal inorganic matter which should be removed are the nutrients, phosphorus and nitrogen. Phosphorus exists after treatment primarily as phosphate, $PO_4^{=}$, and can be removed by anion, or negative ion, exchange. Nitrogen exists largely in the form of ammonium or the nitrate ion, depending upon the sewage treatment it has received. Assuming that nitrogen is in the form of nitrates, it can also be removed by anion exchange.

In addition, much of the remaining organic material, if the sewage is well treated by biological means, can be removed by ion exchange. Phenol can be removed by an anion exchanger. Much of the COD and color, and most ABS is adsorbed on some anion exchange resins. It has also been reported that strongly basic anion exchange resin is able to remove coliform bacteria[4] and virus[5] Therefore, it appears that much of the undesirable material in waste water can be removed by passage through an anion exchange resin.

Previous Studies

In the field of water treatment, cation exchange has been much used for softening and also for iron and manganese removal. Anion exchange is used along with cation exchange in de-ionization. Anionic resins have been suggested for removal of sulfates, sulfides, nitrates, fluorides and oxygen[6].

Abrams and Lewon[7] removed ABS by means of a strong-base anion exchange resin using ABS at relatively high concentrations dissolved in tap water. They were able to elute most of the ABS with a combination of low molecular weight alcohol and dilute mineral acid at 40° to 50°C. The capacity of the resin was high and costs for ABS removal alone were reasonable.

Frisch and Kunin[8] discussed the fouling of anion exchange resins by organics and methods for removing these organic materials from the resins. Caustic was ineffective in removing foulants, but 5 - 10% sodium chloride was effective, particularly at 110° to 130°F (43° - 54°C). Frequent use of low concentrations of brine was more efficient than infrequent use of high concentrations. Strong oxidizing agents were effective also, but damage to resin could result.

Theoretical Considerations

Many materials both natural and synthetic exhibit ion exchange properties. Organic materials including living tissue have these properties, as do many clays. Natural aluminosilicates, called greensands or zeolites, have been used commercially for many years as ion exchange materials. Sulfonated coal has found some use, but more recently synthetic ion exchange materials have been used extensively. Synthetic exchangers are complex and highly varied materials, but they all are insoluble three-dimensional networks which have the ability to exchange either negative or positive ions. Sites exist within the structure where an ion is released and another attached. They behave much like ions in solution in this respect, while remaining in solid form.

The reaction can be represented as follows for an anion exchange when R represents the ion exchange resin and A^- and B^-, two negative ions.

$$RA + B^- \rightleftarrows RB + A^-$$

The reaction is completely reversible as shown by the double arrows. Since this is so, the equilibrium can be expressed by the mass action equation:

$$K = \left(\frac{RB}{RA}\right)\left(\frac{A^-}{B^-}\right)$$

where K is called the selectivity coefficient.

Although the mass action equation is quite useful in explaining what happens, we cannot simply measure a few constants for various resins and predict the outcome of an ion exchange process. Different resins have different affinities for the various ions. There are many complicating factors and the present state of knowledge does not permit accurate prediction of results[9]. There are certain rules of thumb which can be used in a general way, but beyond that, recourse must be had to experiments for each situation. A few general rules are as follows:

1. At low ion concentrations, resins have an affinity for ions of high valence.
2. Exchange increases with atomic number.
3. At high ion concentrations the above effects tend to disappear.

RESULTS AND DISCUSSION

Procedure

Ion exchange resins were used in 50-ml burettes. Flow was controlled with the burette stopcock and measured with a flow meter. Influent was stored in 2-liter polyethylene aspirator bottles with rubber tubing arranged so that watertight connections would be made to the top of the burette, and a head of several feet was available for forcing the flow through the resin. Flow was downward through the bed of resin and was controlled at about 10.5 bed volumes per hour, which would correspond to 1.6 gpm/ft^2 (6.6 ml/min/cm^2). Initial column length was 15 inches (38 cm), corresponding to a resin volume of 33 cm^3.

The influent used was secondary effluent from a laboratory activated sludge unit, receiving raw sewage at conventional loadings. The influent was sand filtered prior to ion exchange.

When the resin bed became exhausted, it was backwashed with flow upwards through the bed. Regeneration with chlorides followed using downflow, followed by a rinse. Samples were taken after passage of each two liters through the resin; usually five such samples could be obtained per run prior to bed exhaustion.

Strong-Base Resin

The most promising resin used was the strong-base anion exchanger, Duolite A-102D, as manufactured by the Diamond Alkali Company. This resin is Type 2 with quaternary ammonium functional groups superimposed upon a cross-linked polystyrene matrix. It is highly porous and comes in bead form.

Results of five runs to exhaustion of this resin are shown in Figure 1, and include about 1600 bed volume of throughput. Individual runs varied considerably, principally due to variation in the composition of the treated sewage, so composites of several runs were found to be more meaningful than individual runs. Variation in results between runs could also have been caused by changes in flow rate and resting of resin between samples. Regeneration was achieved using 2 bed volumes of 10% sodium chloride.

Removals of phosphorus and nitrogen were excellent to over 200 bed volumes of flow in each run. Nitrites broke through somewhat before this, but quantities of nitrite in the treated sewage were small in any case. Significant amounts of organic materials were also removed as evidenced by the removal of color, COD and ABS.

Since chloride exchange was used, chlorides were added to the effluent while other ions were removed. The chloride content in the effluent decreased as the run progressed, and removals of the other ions decreased.

Table 2 shows the average of the influent concentrations which correspond to data shown in Figure 1, and several other figures.

TABLE 2

INFLUENT COMPOSITION, mg/l

	Fig. 1	Fig. 2	Fig. 3	Fig. 5	Fig. 6
Phosphate (as P)	7.6	8.5	11.1	7.6	7.8
Nitrate (as N)	18.4	16.8	13.6	21.3	25.5
Nitrite (as N)	1.06	1.00	2.5	0.76	0.22
COD	33	36	40	26	62
ABS	1.10	0.94	1.65	1.00	0.88
Color (units)	65	58	55	39	34
Chlorides	128	110	310	$\binom{117}{520}$	138

Sulfate content in the sewage had a marked effect on the length of runs. Sulfates were almost completely exchanged for chlorides so that a high sulfate content rapidly exhausted the resin's capacity for other ions.

The capacity of the resin for the length of the run for nine runs to exhaustion ranged from 0.62 Meq/ml (milliequivalents per milliliter) to 1.01 Meq/ml. The lengths of runs were essentially as shown in Figures 1 and 4--runs were carried past phosphate breakthrough, but often were stopped short of total exhaustion. For comparison, the handbook values of theoretical capacity and operating capacity for this resin are 1.4 Meq/ml and 0.8 - 0.85 Meq/ml, respectively.

Weak-Base Resin

A weak-base anion exchange resin, Duolite A-7, also was tried. This resin has a porous phenolic matrix and amine functional groups and comes in granular form.

Results of the average of two runs to exhaustion including 600 bed volumes of throughput are shown in Figure 2. Influent characteristics are shown in Table 2. Regeneration was performed using 2 bed volumes of 1-normal sodium hydroxide followed by 2 bed volumes of 1-normal hydrochloric acid.

Good removal of nitrogen was achieved, but performance on phosphorus was poor. Breakthrough of phosphates occurred early and concentration in the effluent increased rapidly thereafter. Since phosphorus may be the key element in algae control, the use of the weak-base resin was abandoned in favor of the strong-base resin.

The weak-base resin did a much better job in removing organic material. COD and ABS removals were better than those for the strong-base resin, and almost all color was consistently removed. The high affinity for color exhibited by the weak-base resin is probably due to its porosity and ability to absorb acids, as color is commonly believed to be caused by organic acids of high molecular weight. Chlorides were increased while the other ions were decreased as with the strong-base resin, except that chlorides were higher with the weak than with the strong-base resin. This is due to hydrolysis of the HCl from the resin during adsorption.

Regeneration

When resin becomes exhausted, it must be regenerated to remove inorganic ions so that the resin may be used again. Desired characteristics for a regenerant are:

1. An ion which is innocuous in water.
2. An ion which is held less strongly to the resin than ions which are to be removed from water.
3. Low cost.

The chloride ion is one available which most closely approaches these requirements. The cost is low. When used with a strong-base resin, it is quite effective in removing undesirable ions. In low concentrations, it is quite harmless.

However, in high concentrations chlorides can be objectionable. The U.S. Public Health Service Drinking Water Standards[10] limit chlorides to 250 mg/l. This is based on the fact that taste is imparted to water for some people above this concentration. Chlorides can be detrimental to water meant for agricultural use also. Although there are some crops which are sensitive to an excessive chloride concentration, the principal effect of chlorides is to increase salinity which tends to inhibit the osmotic pressure the plant is able to exert.[11] An ion exchange process does not necessarily increase salinity as one ion is exchanged for another.

Unfortunately, Palo Alto sewage, upon which all tests have been run, is high in chlorides. This is apparently due to infiltration of sea water into low-lying sewer lines. The average chloride content at the effluent of the runs shown in Figure 1 prior to breakthrough was just over the 250 mg/l standard for drinking water. Tests on a sewage lower in chlorides concentration than Palo Alto sewage would probably be more favorable. Not only would chlorides in the effluent be lower, but performance in removing other ions would be improved.

Use of Sea Water as Regenerant

One possible economy would be to use sea water as a source of chlorides for regeneration, rather than sodium chloride. A number of runs to exhaustion were made using A-102D with filtered sea water as a regenerant. Initially, the results were good but they gradually deteriorated. The results of one run, at the conclusion of which 1,940 bed volumes of filtered secondary effluent had been put through the resin, are shown in Figure 3. Influent characteristics are shown in Table 2. Influent chlorides were high, but it is believed that poor results were due to other reasons.

Removals of organics as measured by COD and color analysis were much the same as those achieved by this resin using sodium chloride as a regenerant. However, results were poor for removals of inorganics, phosphates, nitrates and nitrites, and ABS. Removals were low and breakthrough occurred early. It is thought that low removals were caused by insoluble compounds of calcium and magnesium, notably calcium phosphate which precipitated out in the resin bed and lowered resin efficiency. Since exchange of inorganics was low, the increase in chlorides was low.

Regeneration of the clogged resin with pure sodium chloride solution restored the full efficiency of the resin. Although the use of sea water as a regenerant was not successful, the possibility of using sea water for a portion of the regeneration remains for future investigation.

Resin Fouling and Restoration

Some organic material is adsorbed on an anion exchange resin and not removed by normal regneration. This material occupies exchange sites, lowering the exchange which can take place, thus fouling the resin. In order to make ion exchange attractive economically, some method must be found for removing this organic material.

Figure 4 shows phosphate removals using one bed of strongly basic Duolite A-102 D. Each curve represents the average phosphate remaining in several runs ending with the quantity of flow shown. The curve for 1,600 bed volumes is identical with that shown in Figure 1. By the time 4,900 bed volumes had been reached, capacity of the resin had been considerably reduced as evidenced by the pronounced shift to the left. Removals before breakthrough were uniformly good. Influent phosphates are shown in Table 3. Resin regeneration was performed using 2 bed volumes of 10% sodium chloride.

TABLE 3

INFLUENT PHOSPHATE (FIG. 4)

Curve	Phosphate (as P) mg/l
1600 bed volumes	7.6
3100	8.3
4900	7.8
6700	6.4

Two methods of resin restoration were tried in an effort to restore the resin to its virgin capacity. The first was the method used by Abrams and Lewon[8] to remove ABS from a fouled resin. This involved using a mixture of five bed volumes each of methanol and 1-normal hydrochloric acid at a temperature of 46°C. The second method was one commonly used in the past to remove silica, a material not removed by chlorides.[12] This treatment used 4 bed volumes of 1-normal sodium hydroxide at a temperature of 38°C.

The curve for 6,700 bed volumes in Figure 4 shows results following both restoration treatments. Considerable improvement is obvious. Unfortunately, two runs to exhaustion between the two restoration attempts failed to indicate how much improvement should be ascribed to each treatment. The first run had high chlorides in the influent and all removals were poor for that reason. One critical sample was lost from the second run. Future work will hopefully show the effectiveness of each method.

High Chlorides in Influent

High chloride content in influent effectively reduced removals--all runs with a chloride content of 200 mg/l or higher were obviously affected In one run shown in Figure 5, when about 4,000 bed volumes had been passed through the resin, the chloride content was relatively low for the first part of the run (117 mg/l) and high (520 mg/l) for the last part of the run. Influent values are shown in Table 2. The resin was strong-base resin. For the first part of the run, removals proceeded in a normal manner. For the last part, however, the action was completely reversed. Chlorides were removed from solution, and phosphates, nitrates and nitrites were added. High chlorides in the influent can have an extremely adverse effect on removals of inorganics. Removals of organics, however, are not affected.

Use of Unfiltered Effluent

All results previously reported were performed using filtered secondary effluent. To investigate the necessity of filtering the influent to the resin, a number of runs were made using unfiltered secondary effluent. The average results of four runs to exhaustion, at the conclusion of which 1,600 bed volumes had been put through A-102 D strong-base resin, are shown in Figure C. The resin was regenerated with 2 bed volumes of 10% sodium chloride prior to each run. Influent values are shown in Table 2.

The results may be compared with Figure 1, showing tests for the same resin and the same amount of influent. Removals of inorganics and ABS were close to those shown in Figure 1, but were slightly inferior. Percentage removals of color and COD were improved but this was, at least in the case of COD, because of filtering action of the resin.

Operating problems were encountered due to the unfiltered influent. It was difficult to maintain the desired flow rate through the resin, and it was also difficult to completely clean the resin by backwash. It is concluded--on the basis of limited laboratory work--that it is desirable to filter the influent, but that no serious harm would result if the filtering were not completely effective. One other possibility would be to remove suspended matter by chemical coagulation and settling before ion exchange. The ion exchange bed in this case would act as a filter to remove any floc carryover.

Waste Disposal

A discussion of water renovation would not be complete without mentioning waste disposal. Waste from ion exchange will consist of backwash water, rinse water, and spent brine, containing small amounts of exchanged ions. Most or all of the backwash water could probably be salvaged by returning it to the influent storage or to a pre-treatment process. Based upon laboratory studies, regenerant would be 2 bed volumes in quantity and rinse water might be as much as 10 bed volumes. From Figures 1 and 4, it appears that each run would amount to about 200 bed volumes. Some economy could be practiced by utilizing the last part of the regenerant for a run for the first part of the regeneration of the subsequent run. In a similar manner the last portion of rinse water for a run could be used for the first part of the rinse for the subsequent run. On this basis the combined quantity of regenerant and rinse water to be disposed of would be about 3% of the total water processed.

The ideal waste disposal point would be the ocean or saline surface or underground waters. It probably would not be desirable to discharge wastes into fresh surface or underground waters. In regions where a convenient disposal point is not available, distillation could be an economical answer considering the small quantity of waste and the possibility of utilizing the distilled water produced.

Cost

In order to evaluate the cost of renovating water, some yardstick or base must be used. The City of Palo Alto purchases its water from the City of San Francisco's Hetch Hetchy System at a cost averaging $65.00 per acre-foot. This is equivalent to 20 cents per 1,000 gallons.

The costs which would be properly charged against a water renovation system would include all treatment costs beyond those of sewage treatment which is normally required in this area. Plant and equipment costs, operational costs, site costs, chemicals, power and waste disposal costs must be included, as well as water storage and transmission facility costs. At the present time only chemical costs can be estimated.

Based on a price for kiln dry salt delivered to Palo Alto at $18.60 per ton when bought in large quantities, a length of run of 200 bed volumes from Figures 1 and 4, and regeneration with 2 bed volumes of 10% solution, the cost for sodium chloride required would be 4 cents per 1,000 gallons. The cost includes the use of some of the product as rinse water and the use of the regenerant twice--the last portion of the regenerant of one run to be used for the first portion of the subsequent regeneration. This cost is for the purchase of the regenerant only.

Costs of resin restoration treatment are uncertain at this time because it is not known what treatments will prove necessary or how often they should be applied. Of the two treatments tried, it is more likely--based on meager test data--that the treatment with sodium hydroxide improved the resin performance rather than the methanol and acid treatment. The cost for the chemicals only for the sodium hydroxide treatment would be 0.4 cents per 1,000 gallons. This cost assumes restoration treatment after each 5,500 bed volumes treated, the point at which the restoration treatment was tried in the laboratory, a cost of sodium hydroxide of $56.00 per ton on a dry basis plus freight to Palo Alto, and use of the chemical twice as previously described.

The methanol and acid restoration treatment would be considerably more expensive due to the high cost of methanol. A recovery system for methanol would probably be justified should this treatment prove necessary.

Resin replacements costs cannot be accurately estimated because resin life is unknown. Based upon resin life in other water treatment processes, it is probable that resin replacement costs will be about the same order of magnitude as the chemical costs previously mentioned--chemical costs for regeneration and restoration treatments.

Future Work

In cooperation with the City of Palo Alto, California, pilot plant studies are now being instigated at the Palo Alto Sewage Treatment Plant for sewage reclamation. There are several advantages to use of the larger-scale equipment. With continuous flow many more cycles can be run to test resin clogging and restoration treatment. The larger-scale study will also be a more effective demonstration to the public.

CONCLUSIONS

1. The strong-base anion exchange resin, regenerated with chlorides, has good potential for water renovation. Algae nutrients, phosphorus and nitrogen, in treated sewage effluent can be reduced to low levels and organic material can also be substantially reduced.
2. Organic material which gradually fouls the resin can be removed by special restoration treatments.
3. Removals by ion exchange resins are only slightly affected by using unfiltered treated sewage, although operational problems are more severe.
4. The process is sensitive to high chloride content in the influent.
5. The use of sea water as a regenerant leads to poor removals due to resin clogging caused by precipitates in the resin bed.
6. Weak-base resin was not satisfactory due to its inability to remove phosphates.

ACKNOWLEDGMENTS

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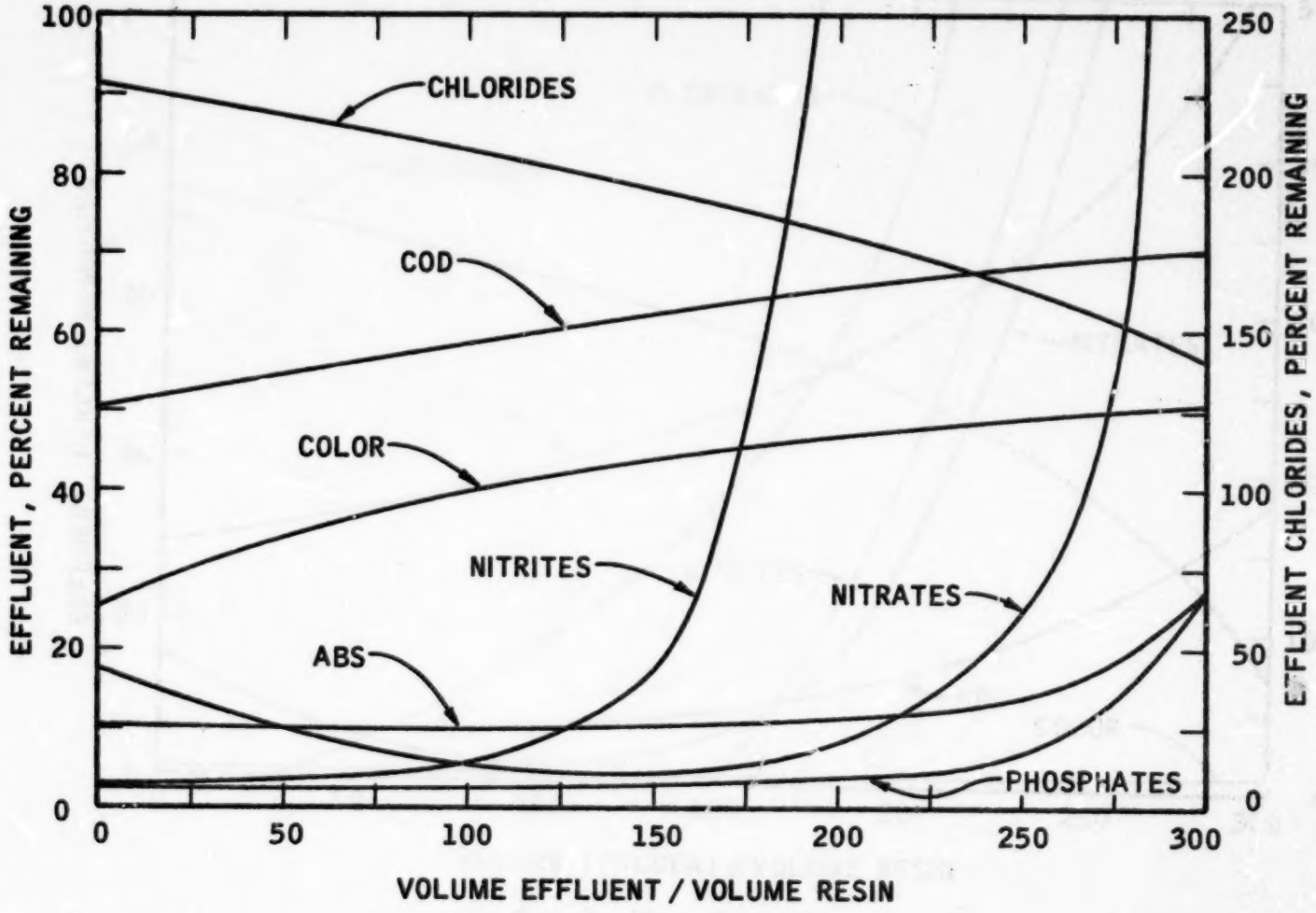

Fig. 1 - Strong-Base Resin

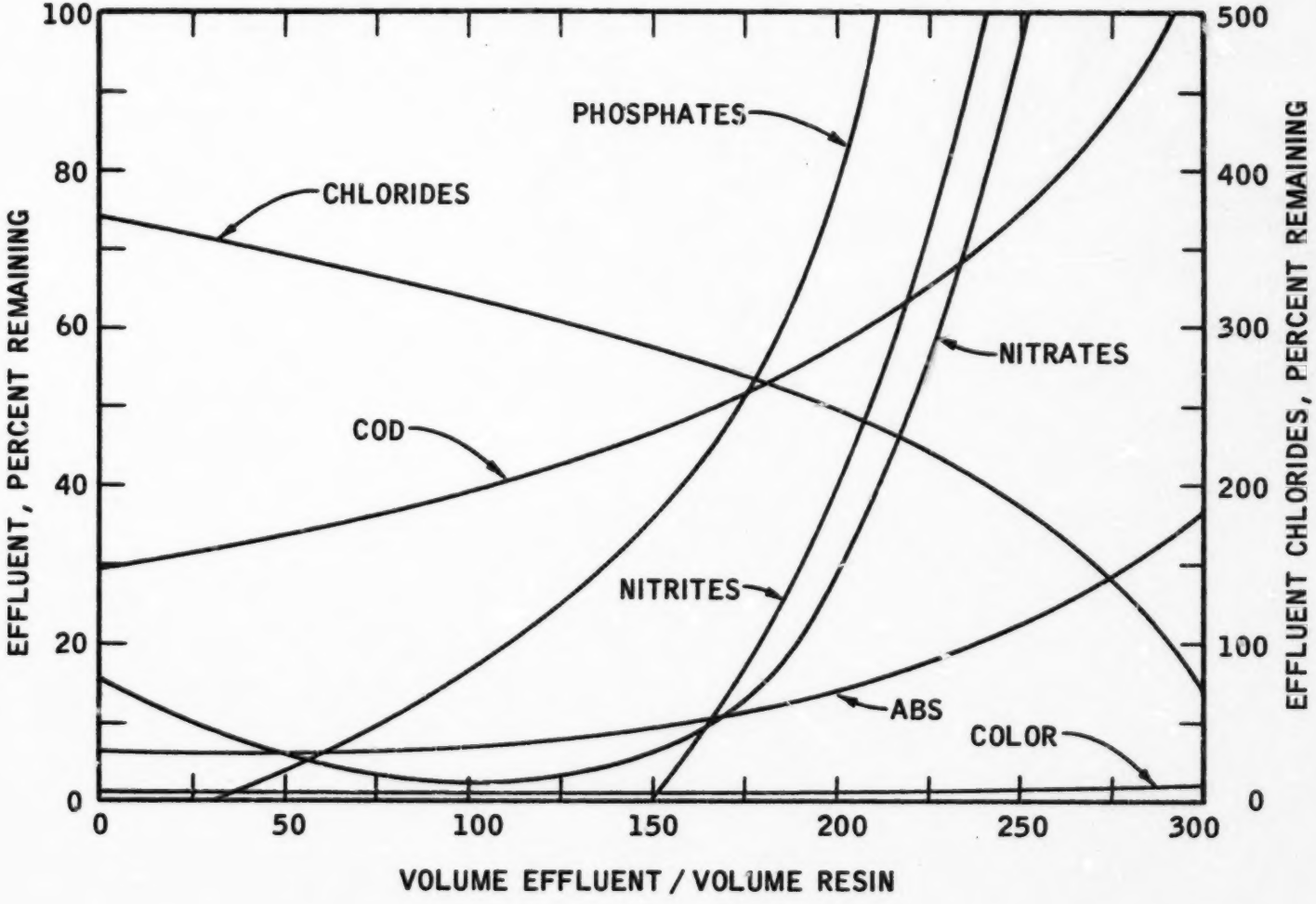

Fig. 2 - Weak-Base Resin

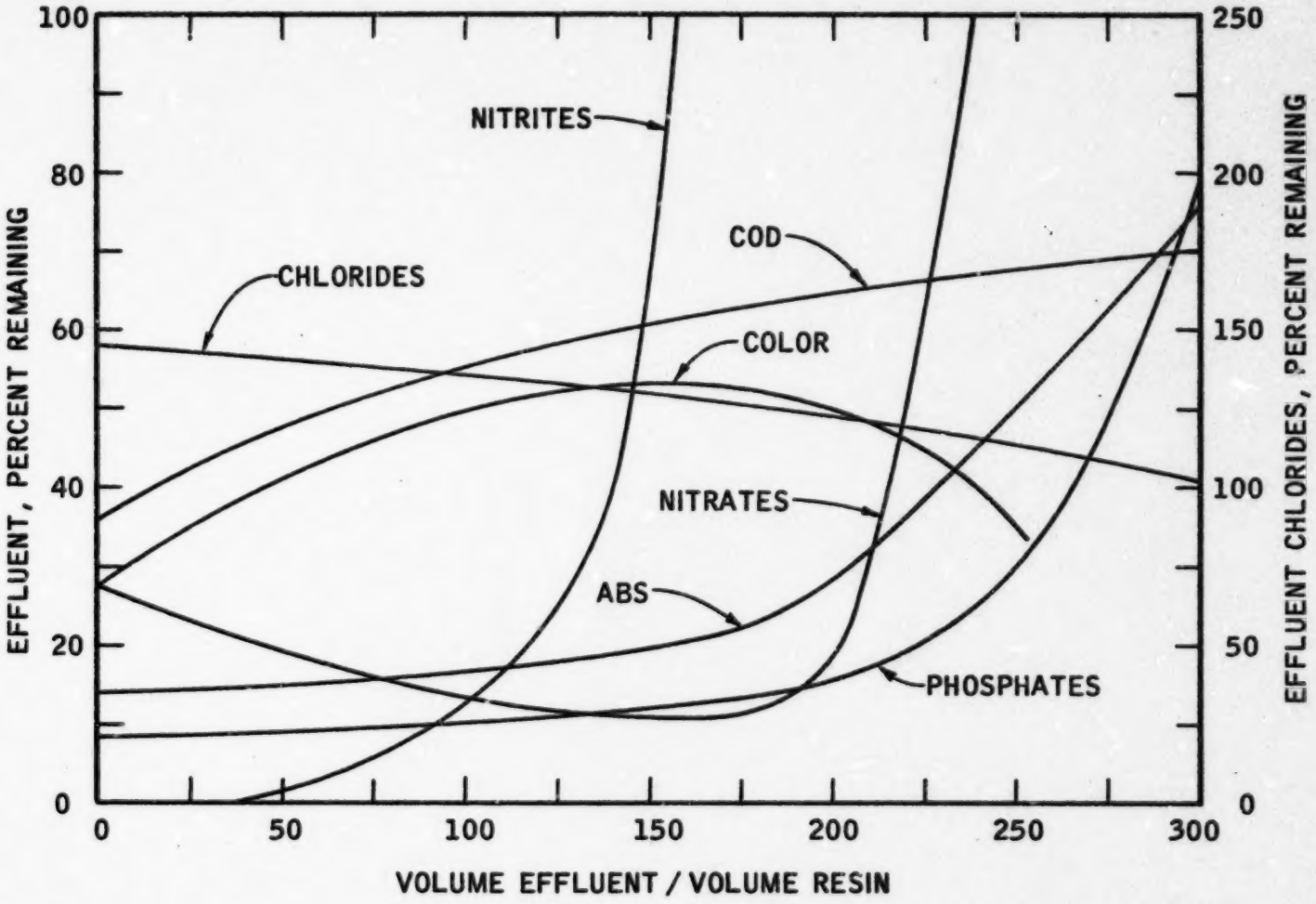

Fig. 3 - Sea Water Regeneration
Strong-Base Resin

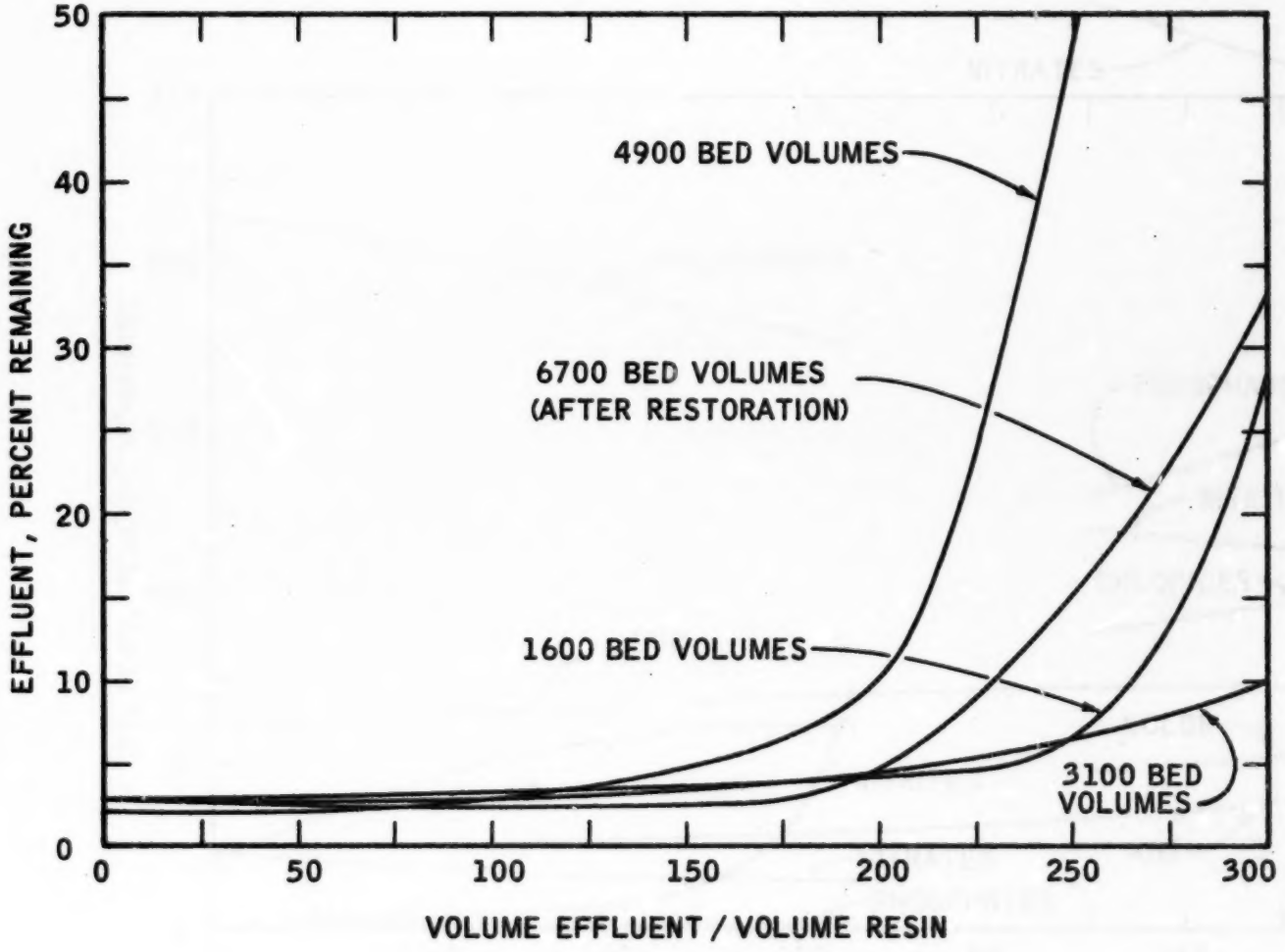

Fig. 4 - Phosphate Removals
Strong-Base Resin

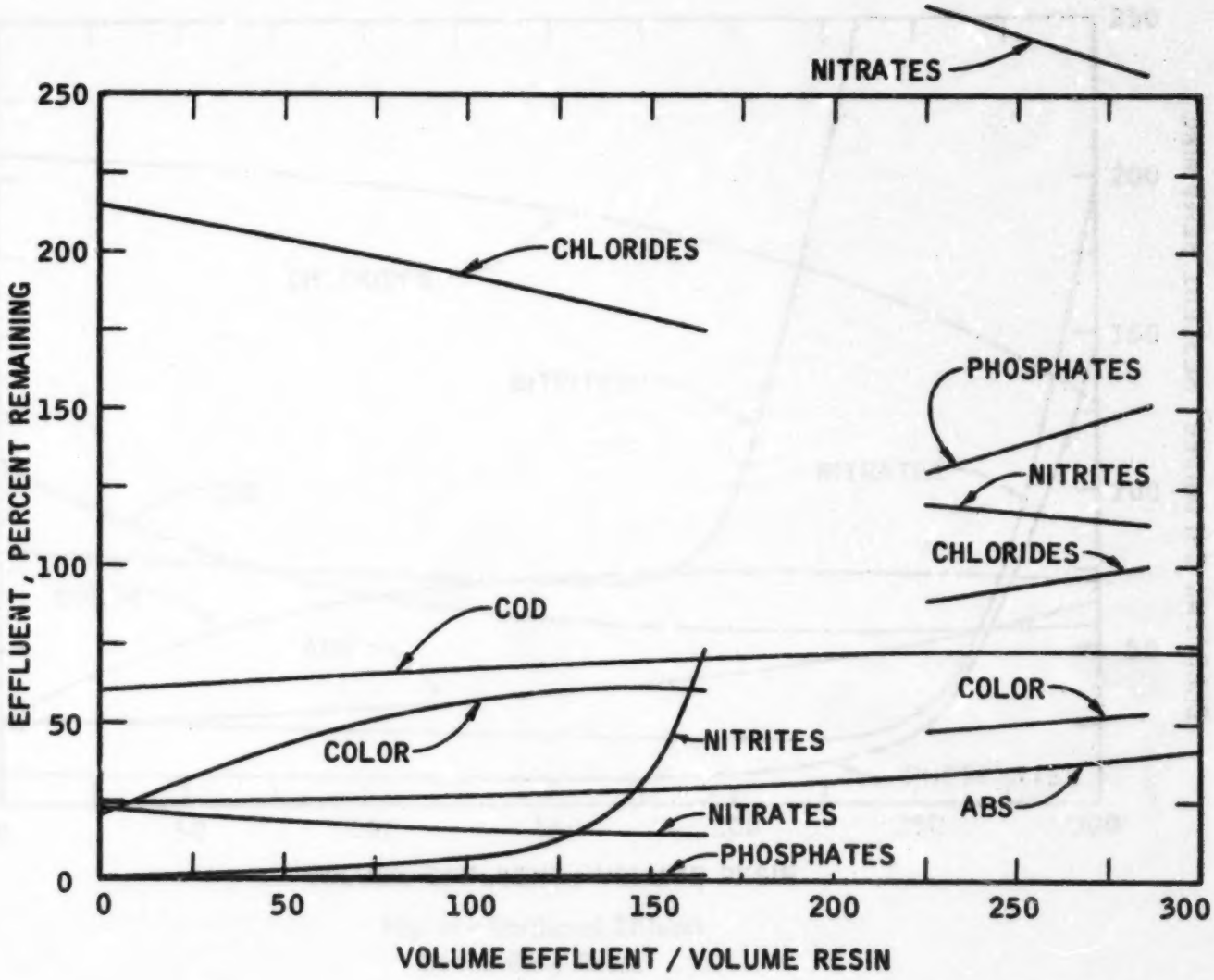

Fig. 5 - High Chlorides in Influent Strong-Base Resin

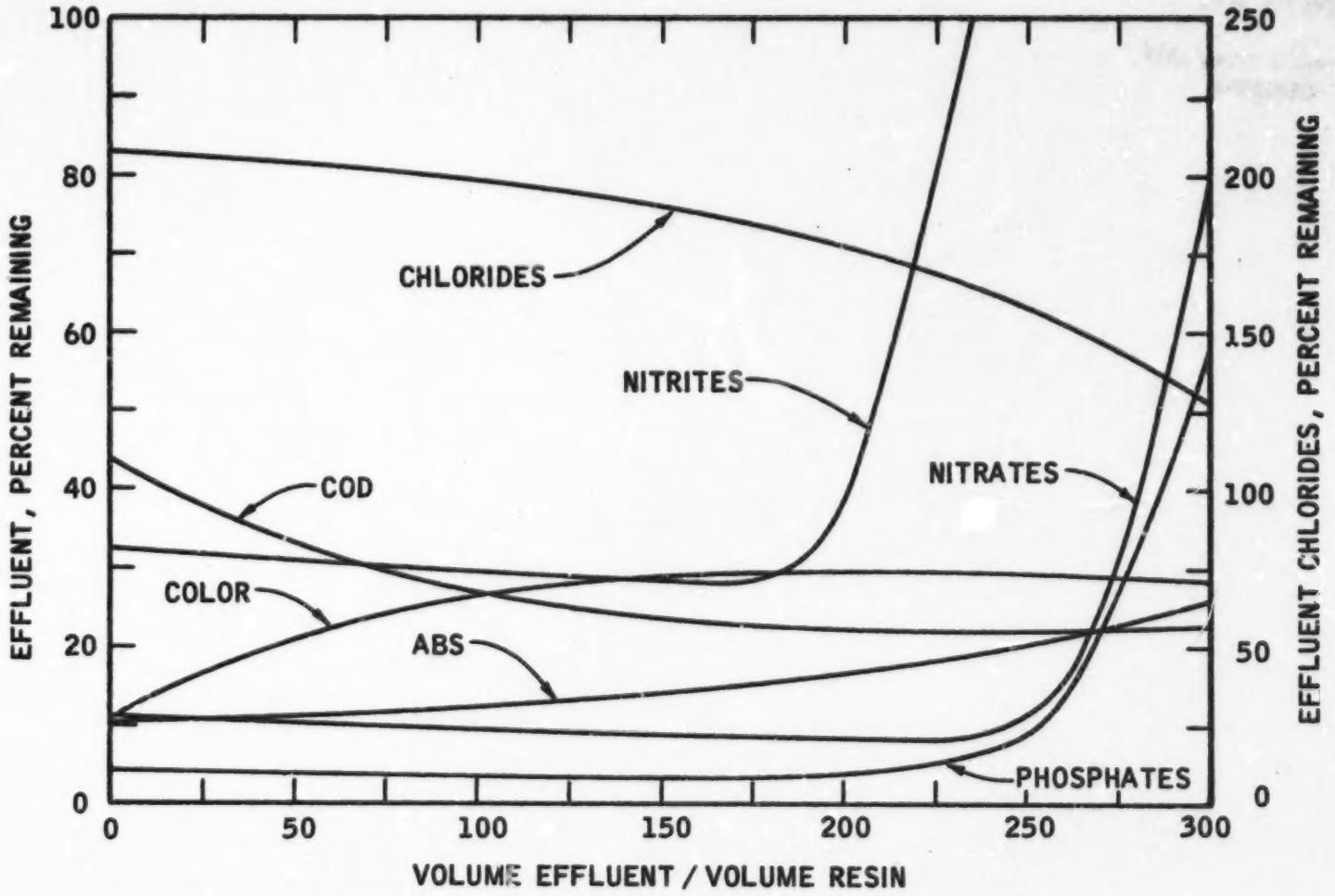

Fig. 6 - Unfiltered Effluent
Strong-Base Resin